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IS 6226-2 (1987): Recommendations for apparatus for chemicals analysis of metals, Part 2: Determination on of sulphur by direct combustion [MTD 4: Wrought Steel Products]

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(Reaffirmed 2002)

Indian Standard

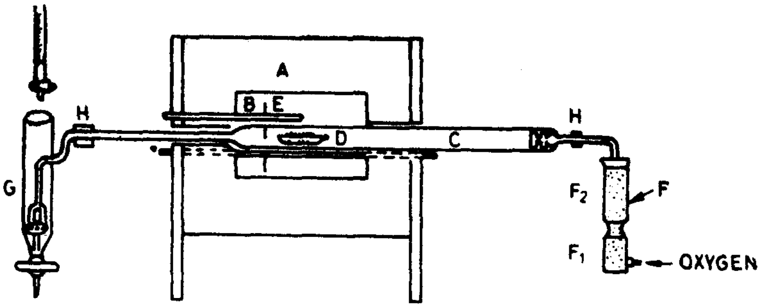
RECOMMENDATIONS FOR APPARATUS FOR CHEMICAL ANALYSIS OF METALS

PART 2 DETERMINATION OF SULPHUR BY DIRECT COMBUSTION

1. Scope — This standard (Part 2) recommends apparatus used for determination of total sulphur in metals, ores and minerals by iodimetric titration after combustion.

2. Apparatus for Determination of Sulphur by the Iodimetric Titration After Combustion

2.1 The apparatus suitable for combustion of the sample in oxygen to form sulphur dioxide (SO_2) and an absorption vessel in which SO_2 titrated is shown in Fig. 1. The apparatus consists of the following parts.



A — Combustion furnace
B — Temperature control
C — Combustion tube
D — Combustion boat
E — Ceramic filter
F — Oxygen purifiers (containing soda asbestos F_1 Anhydrous magnesium perchlorate F_2)
G — Absorption and titration apparatus
H — Rubber stopper and connecting tubes

FIG. 1 TITRATION ASSEMBLY FOR THE DETERMINATION OF SULPHUR

2.1.1 *Combustion furnace* (*A*) — Electric furnace capable of attaining a temperature up to 1 600°C (utilizing rod shaped silicon carbide heating elements is suitable).

2.1.2 *Temperature control* (*B*) — To ensure the reproducibility of results, an automatic temperature controller is used.

2.1.3 *Combustion tube* (*C*) — Tube of refractory type as a crystallized alumina of low sulphur content which can withstand temperature up to 1 600°C and having internal diameter of suitable size to fit in the furnace (a suitable size is 2 to 5 cm in dia and length of 85 cm) so that exist end extends approximately 15 cm beyond the furnace.

2.1.4 *Combustion boats and covers* (*D*) — Refractory boats and covers of very low sulphur (approx size 9 to 10 cm). These are pre-ignited at 1 100°C for 15 minutes and stored in a desiccator prior to use.

2.1.5 *Ceramic filters* (*E*) — Porous ceramic filter is introduced after the combustion zone to trap any metal oxide dust from the gas stream.

2.1.6 *Oxygen purifiers* (*F*) — Oxygen is passed from the cylinder through two pressure reduction valves to provide an even flow which is then passed through absorption tower containing soda asbestos and anhydrous magnesium perchlorate.

2.1.7 *Absorption and titration apparatus* (*G*) — Apparatus consisting of an absorption and titration vessel of appropriate volume and of suitable shape to effect complete absorption of sulphur dioxide.

2.1.8 *Rubber stopper and connecting tubes* (*H*) — Preferably silicon rubber essentially free from sulphur may be used as stopper tubes and connecting tubes.

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, SMDC 2 [Ref : Doc : SMDC 2 (3019)]

Adopted 22 December 1987 © December 1988, BIS Gr 1

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EXPLANATORY NOTE

In various Indian Standards relating to Methods of chemical analysis of metals, alloys and ores, details of apparatus, such as apparatus for carbon and sulphur determination had to be repeated. In order to avoid this repetition need was felt for the standards which include the details of apparatus required. This standard describes the detail description of apparatus required for determination of sulphur. The apparatus details for determinntion of carbon is given in IS : 6226 (Part 1)-1971 'Recommendations for apparatus for chemical analysis of metals'.